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INTRODUCTION

This eighth semi-annual report from the Interdisciplinary Materials Research Program at Rensselaer Polytechnic Institute contains a brief description of the studies being carried out under each project supported by the grant and the progress realized during the report period. No research projects were initiated or terminated during this time.

Occupancy of the new Materials Research Center which is being financed by a 1.5 million dollar grant from NASA was begun during this report period. This new facility will allow the entire program to again be housed under one roof. On March 3, 1965, Dr. R.G. Folsom, President of Rensselaer Polytechnic Institute, presented a paper on the Educational and Industrial Impact of the NASA Materials Research Center at Rensselaer at the NASA University Program Review Conference at Kansas City. This talk highlighted the advantages accruing to both industry and education because of these facilities, and will be published in the proceedings of the Conference.

RESEARCH PROJECTS

Mechanical Properties of Polymers 470.05

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A. Fracture of Polymers

The fracture theory for semi-ductile viscoelastic media, as described briefly in the previous report, has been verified experimentally for creep loading conditions. This theory allows a quantitative prediction of the creep failure stress-time to break relation from constant strain rate failure data. Consideration is now being given to cyclical fatigue loading conditions.

Experimental results obtained thus far have corroborated the theoretical aspects of this study. It has been established that the failure of amorphous, glasslike high polymers, in uniaxial loading, can be treated from a uniform and consistent point of view with a single criterion of incipient criticality for failure, regardless of the uniaxial bulk loading history of the sample. (Supported by the Institute of Paper Chemistry).

B. Polymer Network Mechanics

Numerical solutions to the proposed theory have been obtained for the fiber network problem. These results represent, quantitatively, the relationship between structural parameters of a piecewise continuous array of structural elements and the macroscopic continuum which the array compose. Experimental verification of the theory and its various ramifications are now being considered.

The application of the theory to the prediction of constitutive equations, for elastomeric networks at large deformations, is currently under investigation. (Supported by the National Science Foundation).

C. Viscoelastic Properties of Polymers

A study of the effects of solvent environments on the mechanical properties of high polymers has indicated a need for simultaneous measurement of swelling and absorption phenomena as a function of environment (solvent, time, temperature). Accordingly, and apparatus is now being designed to obtain these data. In addition, microscopy studies of the effect of solvents on various polymers, as a function of stress and environment, have shown that the effect of prior surface treatment has a marked influence on the resistance of a polymer to solvent attack. Further work is in progress to allow more quantitative observations to be made.

The dynamic and dielectric spectra studies are being continuued. The dielectric cell, specifically designed to measure the dielectric properties of solid polymers from $-180^{o}C$ to $+100^{o}C$, has been successfully used and the experimental study of dispersion phenomena in polyvinyl alcohol-acetate copolymers is in progress.

D. High Pressure Infrared Spectra

The intensity diminution phenomenon of the symmetric and asymmetric methylene stretching modes is now being extended to polymers such as

polytetrafluoroethylene, polyvinylidenechloride, and polychlorotrifluoroethylene. Theoretical and experimental studies on these polymers should yield information which is of significance to an understanding of polymer deformation resistance.

A Study of the Interaction of Dislocations with the Discrete Second-Phase Particles in Dispersion-Strengthened Alloys

470.09

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The objective of this program has been to delineate the effects associated with the presence of a distributed second-phase in a crystalline matrix by both theoretical considerations and experimental observations.

During this period, research efforts have centered principally upon experimental observations utilizing both replication and thin film transmission electron microscopy techniques. The specific areas investigated include:

A. Recovery and Recrystallization Kinetics in Two-Phase Systems

It has been well established that the presence of a distributed second-phase inhibits grain boundary migration, particularly secondary recrystallization in dispersion-strengthened alloy systems. Unfortunately, however, the mechanism by which this retardation occurs has never been delineated. Utilizing hot stage transmission electron microscopy and ciné and sequence recording techniques, studies of the rate of substructural recovery of cold worked structures and the interaction of migrating grain boundaries with second-phase particles are continuing. The alloy system investigated is the Al-Al_2O_3 SAP-type alloy. This alloy has afforded direct observation of these kinetic processes without the concomitant change in distributed phase structure which usually accompanies these high temperature processes. In particular, the thermal effect on the cold worked structures can be described in terms of two distinctive mechanisms. At approximately 0.5 of the absolute melting temperature localized shifting of the individual dislocation boundaries in the cell walls produce change in relative cell-to-cell misorientation. At higher temperatures, cell wall migration and resultant growth occurs. The mechanism of cell wall migration is

currently being studied in detail.

B. Work Hardening Behavior in Precipitation-Hardened Alloys

The role of the interface structure upon dislocation distributed phase interactions appears upon the basis of theoretical considerations to be of prime importance in the development of substructure during deformation. Previously in the program, the substructural development in alloys containing noncoherent second-phase particles was studied. At the present time, the substructural development in an Al-Ag precipitation-hardened alloy is being followed by means of transmission electron microscopy. During this period, the change in substructure with rolling deformation in the solid solution treated condition has been defined. Currently, the effect of the precipitate phase upon the worked structure is being studied. In this alloy system, the matrix-particle interface structure can be varied by means of suitable heat treatment. Thus, the effect of precipitate and its structure upon work hardening is to be defined.

C. Fracture Behavior

The influence of a distributed phase upon fracture mode and crack propagation is being studied utilizing the Al-Al_2O_3 SAP-Type alloy as a model alloy system. Replication studies of the fracture surfaces of these alloys broken in fatigue, appear to establish the mechanism by which dissolved gasses, particularly hydrogen, precipitate from solid solution due to the state of elastic stress in the aluminum matrix, due in turn to the constraint of the distributed second-phase. This study is now completed and is being prepared for publication. Extension of this program to the iron hydrogen system is now in progress.

D. Theoretical Studies

Theoretical studies of dispersion-strengthening based upon dislocation theory are progressing on a continuing basis.

Mechanisms of Solidification 470.10

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Contrary to what would be expected from conventional theory, supercooled bismuth has been nucleated by cintrifuging without vibration. The resulting dynamic supercooling is decreased reproducibly by increasing centrifugal rotational speed.

It was felt that a theory which would explain this unexpected

result would do much to clarify the whole mechanism of nucleation in liquid metals.

It was found possible by the use of dimensionless analysis to develop a relationship between the ratio of the temperature of dynamic nucleation and of static nucleation and the mechanical energy added to the system. This relationship extended to systems as diverse as sinusoidal vibration and centrifuging.

It has also been demonstrated that in the audio range of vibration small amounts of vibrational energy actually hinder nucleation, while above a given energy level, the vibrational energy increasingly facilitates nucleation.

The nucleation behavior of these pure metals and alloys when various types of mechanical energy are added to the system is being used to formulate a general theory of nucleation.

Ultrasonic Research 470.11

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Current attention is focussed on an investigation of the third order elastic constants of LiF using the same techniques as were employed earlier with success for NaCl and KCl. The LiF can be expected to show a rather different pattern because of the decreased ratio of cation to anion size. Moreover we may not be able to invoke the Cauchy relations for the third order constants with the same confidence.

With a view to the future we are considering measuring the elastic constants of certain nickel-iron alloys over an extended temperature range. This investigation is part of an interdisciplinary effort to explore the changes in elastic and X-ray parameters around the Curie point. Another project under consideration for future development is a 3-phonon study wherein two intersecting ultrasonic beams will be used to generate a third beam by virtue of the anharmonic properties of the transmitting medium.

Ultrasonic Pulse Interferometry 470.12

Senior Investigator: S. Katz, Ph.D.
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Solid-solid phase transformations in several materials are being studied with a high-pressure cell using diamond anvils and infrared spectrophotometric and visual microscopic techniques. During the past six months considerable progress has been made in a study of the pressure-induced transition of $CaCO_3$ from the hexagonal to the higher-density, orthorhombic structure (calcite $\rightleftarrows$ aragonite). This transition is of special interest because of the geologic occurrence of the two forms, and because of the importance of shear and high temperature in determining transition direction and reaction rate. In the course of this study, the cell was calibrated by using well-known solid-solid transition points, including AgI at 2.5 kb, $AgNO_3$ at 9.8 kb, RbCl at 10.8 kb, and KCl at 19.3 kb. With this information, radial pressure gradients have been calculated and a quantitative interpretation of infrared spectra is now possible. One of the results of the calibration is the discovery that the true pressure at the anvil centers is nearly twice the nominal pressure (force/area). The applicability of this calibration to the $CaCO_3$ system has been demonstrated by diluting KBr specimens with varying amounts of $CaCO_3$ without affecting the calibration.

As a result of a change in site symmetry with respect to the carbonate ($CO_3^=$) ion, the infrared absorption in calcite and aragonite differs in two important respects. In aragonite, the ν_1 symmetric stretching mode is active, and the ν_2 symmetric bending mode shifts toward the red approximately 25 cm^{-1}. Since the magnitude of the shift of the ν_2 fundamental results in overlapping spectral bands, a method of separating the spectral lines, devised by H. Stone (Jour. Opt. Soc. Amer., 52, 998-1003, 1962) and written into a computer program, is being used.

To determine quantitatively the percentages of calcite and aragonite present in a sample, the ratio-method of analysis (Potts, W.J. Jr., Chemical Infrared Spectroscopy, v. 1, Techniques, John Wiley and Sons, New York, 1963) is also being used in conjunction with the program. Compositional values in unknown specimens can be determined with considerable precision to within 1 percent. A paper describing this method of quantitative analysis and its application to this and other types of compositional changes is in preparation. The results obtained so far suggest that aragonite is being produced above a transition pressure of 0.9 ± 0.3 kb at 25°C and that the reaction is partly irreversible, with powdered material reverting to the stable form in greater amounts than single crystal specimens.

In addition to completing the above study, it is planned to introduce temperature as a variable by a heating cell which has been constructed and to study other materials. Among such materials are germania and the rock-forming silicates. Synthetic magnesium silicate and germanate have been prepared. These form an isomorphous solid-solution series, the silicate end of which is generally thought to be a major constituent of the upper mantle of the earth.

The Relation of Molecular Structure and Intermolecular Action in Flow of Polymers and Polymer Dispersions as a Function of Temperature and Shear Rate 470.15

Senior Investigator: W.H. Bauer, Ph.D.
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The objective of the research is the study of the relation between molecular structure and flow properties of polymers of the low molecular rubber type with the objective of relating the use properties to the molecular structure.

In the past six months pressure capillary measurements have been made on a variety of polymers selected for the influence of the carboxyl group, molecular weight and molecular weight distribution. Molecular weight and distributions have been tested by various methods. Flow properties have been determined at a number of temperatures.

In the immediate future it is planned to continue the measurement of flow properties at high rates of shear and at various temperatures for the remainder of the members of the polymer series selected for study.

High Temperature and Mechanical Metallurgy 470.18

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A. High Temperature Oxidation of Cobalt:

The doctoral thesis of Mr. J.L. Solomon has been completed, presented, and accepted. Samples of cobalt metal were oxidized in dry oxygen at atmospheric pressure, and weight change due to formation of oxides was continuously recorded in special apparatus. Oxidation constants were computed from the experimental data and were plotted as a function of 1/T. This Arrhenius type of rate plot indicated that the kinetics for cobalt oxidation were controlled by different activation processes at different temperatures. For $T < 700^{\circ}C$, Q_3 = 31 kcal/mole. For $700^{\circ} < T < 1120^{\circ}C$, Q_2 = 48 kcal/mole; and for $T > 1120^{\circ}C$, then Q_1 = 29 kcal/mole. Q_1 and Q_3 were found to be dependent upon the diffusion of Co through CoO, while Q_2 was found to be dependent upon the diffusion of Co through Co_3O_4. The diffusivity of Co through Co_3O_4 was then calculated and found to be equal to:

$$D = 6.75 \times 10^{-3} \exp(-48{,}243/RT) \text{ cm}^2/\text{sec}$$

The change in kinetics at the Curie point was explained by a magnetic energy interaction between the Co^{+2} ions in the lattice. Evidence of preferred orientation in the oxide was found by x-ray diffraction techniques.

B. Relations Between Hardenability, Impact Behavior, and Microstructure

Steel corporations have provided some forty-odd samples of alloy steels with slightly differing chemical compositions. All steels were heat treated so as to give virtually identical tensile properties, and the heat treatments were accomplished in identical manners as regards quenchant and size of section. However, there was sufficient difference in chemistry, from steel to steel, to allow comparatively large changes in hardenability, as judged by "Ideal Critical Diameter".

Excellent correlations between the "Ideal Critical Diameter", (As calculated by several different methods), the 15 ft-lb transition temperature, and microstructure have been determined. A technical paper summarizing results is currently being prepared.

C. Effects of Retained Austenite on Fracture Characteristics

Two alloy steels (4340 and 52100) have been heat treated so as to have several different degrees of retained austenite. The effects of this retained austenite, which is face-centered-cubic, on the cleavage and ductile fracture characteristics of the steels, which are mostly a body-centered-cubic matrix, is being studied in detail.

Low Temperature Physics 470.19

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A. Ultrasonic Attenuation in Superconductors

The ultrasonic attenuation measurement is being used to investigate the gap in the electronic energy levels in superconductors. In addition an amplitude dependence of the attenuation in superconducting lead has been found and is being further studied. An explanation for this effect proposed here and partially worked out by W.P. Mason of Bell Telephone

Laboratories is the following: Dislocation motion in the normal lead crystal is heavily damped by electron interaction. Under these conditions the amplitude of vibration of the dislocation lines under the action of the ultrasonic wave is small and only an amplitude independent attenuation is contributed. When the metal becomes superconducting, however, a certain fraction of electrons, dependent upon temperature, are no longer able to interact with the dislocations. The amplitude of the dislocation motion then increases and eventually reaches sufficient levels to break away from pinning points and contribute an amplitude dependent term. Of current interest are studies of the dependence of this effect upon purity, plastic deformation, and temperature. All of these will serve as tests of the above hypothesis.

Preliminary studies in superconducting mercury have also been carried out and will be followed up. Thus far there is an indication of an energy gap in much better agreement with measurements by other techniques but no indication of the amplitude dependence found in lead.

B. Superconductivity and Lattice Defects

Work in this area since the last report has concentrated upon obtaining better knowledge of the defects introduced by low temperature deformation and their influence on superconducting properties in lead, indium, and thallium. In these cases no resistance minimum is created such as was found for alloy samples earlier. The resistive transition to superconductivity is significantly broadened for lead but no such effect is observed in indium and thallium. Both lead and thallium have been observed to have a strongly enhanced longitudinal magnetoresistance in the normal state following deformation. Preliminary results indicate that this effect anneals with the dislocations rather than point defects.

Future studies will include magnetic moment and thermal conductivity measurements on the pure materials and alloys in addition to further resistivity measurements. Of particular interest are the roles played by crystal structure and the Ginsburg-Landau K parameter in the sensitivity of these properties to deformation.

Ceramics Research 470.20

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A. Current Research

(a) A detailed microstructural study of the naturally occurring flexible composite material, itacolumite or flexible sandstone, was made to (1) ascertain the role of mica in this predominantly quartz

grain material and (2) to deduce the mechanism of formation of the composite. We conclude: (1) that mica is the primary bonding agent in our samples; (2) that mica plays only a minor role in the flexibility; (3) that a similar composite might be made synthetically by leaching the proper starting material under pressure; and (4) that the "loose grain" mechanism of bending on this material is confirmed by quantitative microstructural measurements.

(b) For the period September 1964 to March 1965 the following research has been done on the vaporization of sapphire and ruby in dry hydrogen.

(1) The study of the reduction of sapphire crystals in the temperature range 1500°C-2000°C and subsequent growth of sapphire whiskers has been continued. It is expected that quantitative results of this study will be available this summer.

(2) The curious behavior of Cr in rubies during reduction is still under investigation. Present evidence suggests that Cr does not form lower oxides like Al in a reducing atmosphere. The workers are presenting a paper on the reduction of Cr_2O_3 at the May Ceramics Society Meeting in Philadelphia.

(c) A mathematical model was set up for reduction of two component oxide solid solutions. This model displayed the segregation effect which is observed during reduction in such systems. This model was presented at the March Meeting of the AIME in Chicago.

B. Future Research

The Al_2O_3 reduction study will be continued. The program of the new staff member, Dr. C. Willingham, who will take over the ceramics research, will determine the future direction of this area.

Dispersion-Strengthened Materials 470.23

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The investigation of the strengthening mechanism of internally oxidized silver-magnesium alloys is being continued. It had been found that the increase in hardness and lattice parameter of internally oxidized alloys over those of non-oxidized alloys is the greater, the lower the temperature of oxidation. When alloys oxidized at a low

temperature (say 600°C) are annealed at a higher temperature, e.g. 900°, their hardness and lattice parameter decrease to a value characteristic for alloys oxidized at the higher temperature. A theory postulating very small precipitate particles, whose size depends upon the temperature of oxidation, was developed. The persistent effort to actually show particles or the strain effect of these particles by transmission on electron micrography in the internally oxidized alloys is being pursued in order to confirm the theory which originally was based purely on macroscopic properties. The first transmission electron micrographs actually showing D-shaped lobes as an indication of particles straining the lattice have just been taken.

The work on the creep properties of the internally oxidized alloys is continuing. These tests are quite time consuming because good values for creep rate in alloys in which the creep rate is very low, can be obtained only if the experiments are continued for a long time.

Single crystals of silver-magnesium alloys of two compositions have been grown. The techniques of producing tensile specimens of these alloys, internally oxidizing the specimens and determining the critical resolve shear stress and the stress-strain curve of the specimens are being developed.

Replicas of ice specimens produced from a colloidal suspension of silica in water indicate that the dispersion of silica in ice is reasonably uniform, if the process of freezing is carefully controlled. The production of tensile specimens and creep and tensile tests on these specimens in the temperature range -20 to -30°C are under way.

Metallic Dissolution 470.25

Senior Investigator:	N.D. Greene, Ph.D. Associate Professor of Metallurgical Engineering
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The purpose of this program is to determine the basic corrosion and electrochemical characteristics of iron and iron alloys (steels). Although the corrosion behaviors of iron and commercial steels have been empirically determined by conventional corrosion tests, few studies have been devoted to determining the effect of metallurgical variables on electrochemical properties. Composition and/or structure have a pronounced effect on chemical behavior as evidenced by the wide variations observed between nearly identical samples.

Current research includes extensive corrosion testing of carbon and low alloy steels in acid media. Upon completion of these measurements, multiple correlation analyses will be performed to determine relationships between corrosion behavior, chemical composition and

alloy structure. Also, similar analyses will be applied to electrochemical data obtained from anodic and cathodic polarization experiments.

Nuclear Magnetic Resonance Research 470.26

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The study of the Na^{23} nuclear magnetic resonance in Rochelle Salt is nearly complete. Accurate values of the Na^{23} quadrupole coupling constant have been obtained in both the ferroelectric phase, and in the high temperature paraelectric phase. The change in the coupling constant has been analyzed in terms of changes in atomic positions which occur at the Curie temperature. This same technique has recently been used to study the phase change in deuterated Rochelle Salt in another laboratory, and a comparison of the results is enlightening. The results of both experiments indicate that the only changes in the crystal are a movement of one water molecule and one hydroxyl ion. Furthermore these displacements are found to be larger in the deuterated salt, as expected, since its spontaneous polarization is greater than that of the undeuterated salt. In addition, recent neutron diffraction studies indicate that these same two groups, the water molecule and hydroxyl ion, are the only two units in the crystal that move at the Curie point. Thus it is felt that the structural changes that take place in Rochelle Salt are now well understood.

X-Ray Scattering 470.28

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The temperature dependence of the x-ray Debye Temperature in chromium has been studied down to liquid nitrogen temperature. The data are being analyzed and compared with the elastic constant Debye temperature data of D.I. Bolef and J. de Klerk (Anomalies in the Elastic Constants and Thermal Expansion of Chromium Single Crystals; Phys. Rev. 129, 1063, (1963).) and the specific heat Debye temperature data of K.

Clusius and P. Franzosini (Atom-und Elektronenwarme des Chroms zwischen 14°K und 273°K; Z Naturforsch, 17a, 522, (1962)). A liquid helium cryostat which will extend these x-ray studies down to approximately 3.5°K will be in operation in the near future.

The paper entitled, "The Crystal and Molecular Structure of Ru-SO_2 Complexes: I, $[Ru^{II}(NH_3)_4SO_2\ Cl]\ Cl$" by Lester H. Vogt, Jr., J. Lawrence Katz and Stephen E. Wiberley has been accepted for publication by the Journal of Inorganic Chemistry. Work on the bromo-bromide and related complexes is in progress.

A new study has been initiated on the crystal structure of $LaCl_3$ in order to obtain more significant results then previously available.

Thermal Properties of Polymers 470.29

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A. Thermal Conductivity

A series of measurements of thermal conductivity of polystyrenes of different molecular weights have been completed. The results confirm earlier conclusions based on theoretical calculations and measurements on polyethylene. A detailed report on this work is being prepared for publication.

Experimental work on thermal conductivity anisotropy in oriented polymers in underway. Results obtained to date are only fragmentary and inconclusive.

B. Structure

Structural changes in linear polyethylene on cold-drawing have been studied using polarized light microscopy of microtome sections cut from drawn and undrawn material. The optical bands or rings in the undrawn spherulites change their orientation on drawing in a manner indicating that the crystal-slip mechanism postulated by Hansen and Rusnock is a primary deformation mechanism in cold drawing of polyethylene. A paper reporting the details of this work is being prepared for publication.

Work is continuing on patterns of crystallization in bulk polymers and deformation of structure on cold-drawing.

Glass and Non-Metallic Materials 470.30

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The objectives of this work are twofold: (a) to correlate structures and properties of glasses and other non-metallic solids, and (b) to prepare new materials and study their properties.

During this period, research has been carried out on (1) semiconducting glasses, (2) ceramic-metal interfaces, (3) glass-crystalline oxide composite materials, (4) electrical conduction in high resistivity glasses, and (5) vapor deposition of oxide glasses. These projects will be continued in the next six months.

Vibrational Spectroscopy Inorganic Substances in the Vapor Phase 470.31

Senior Investigator: S.C. Wait Jr., Ph.D.
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Research Staff: G. Kelly, B.A.
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Investigations of the infrared spectra of inorganic substances in the vapor phase are being undertaken for the purposes of making vibrational assignments and normal coordinate analyses and investigation ionic interactions and models to represent such interactions. An infrared cell suitable for use at elevated temperatures-up to at least 750°C. has been constructed and preliminary volatility studies on inorganic nitrates are being carried out. In this respect a continuing literature survey of spectral studies of inorganic substances in the vapor phase is in progress and may be submitted for publication. Future plans include direct spectral investigations of nitrates. Preliminary studies will center on the alkali metal nitrates, but if volatility of these is too low, emphasis will shift to more "covalent" nitrates, e.g. copper nitrate and other nitrates recognized as being more volatile.

Irreversible Thermodynamics of the Solid State of Linear High Polymers

470.32

Senior Investigator: B. Wunderlich, Ph.D.
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Thermal measurements on linear high polymers have been hampered in the past because of the irreversible nature of most processes occurring in these materials. In the past, it was shown in our laboratory that: (1) these difficulties can be overcome by using "zero entropy production" measuring conditions and (2) it is possible to produce extended chain equilibrium crystals.

During the last six months, the specific heats of equilibrium crystals of polyethylene have been measured using the Perkin-Elmer differential scanning calorimeter, purchased under the present grant.[1] A study of copolymers grown under equilibrium conditions was completed using both the Perkin-Elmer DSC-1 and the previously acquired du Pont DTA-analyzer.[2] In addition, fast heating experiments reported on in the previous report were continued.[3]

For the next 6 months, it is planned to finish the major study of superheating of polymethylene and poly(tetrafluoroethylene) and to start further investigation of crystallization and melting of polymer crystals.

1 "Specific Heat of Polyethylene Single Crystals: by B. Wunderlich (accepted J. Phys. Chem.)

2 "Thermodynamics of Crystalline Linear High Polymers. V. Extended Chain Copolymers of Polyethylene" by B. Wunderlich (to be printed in Polymer Preprints, Am. Chem. Soc., Spring Meeting 1965, and submitted to J. Polymer Sci.

3 "Zeitabhängige Vorgänge des Kristallisierens und Erstarrens bei Linearen Hochpolymeren" (accepted Kunststoffe)

Ultra-Low Temperature Solid State Physics Research

470.33

Senior Investigator: G.L. Salinger, Ph.D.
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The purpose of the project is to investigate magnetic interactions in dilute magnetic salts at low temperatures. It is proposed to measure the thermal conductivity in the ordered phase in order to determine magnetic coupling constants.

This six months has been spent in preparing the low temperature apparatus. The He^3 refrigerator has been designed and the measuring apparatus has been built. Hydrated crystals of cesium, magnesium nitrate and chrome alum for use as thermometers and magnetic refrigerators have been grown.

In the next six months it is hoped that the apparatus will be completed and tested. Experimental samples will be prepared.

Thermal Decomposition of Inorganic Coordination Compounds — 470.34

Senior Investigator: R.A. Bailey, Ph.D.
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Research Staff: W. Tangredi, M.S.
Graduate Assistant

The objective of this study is to investigate the manners in which coordination compounds containing organic ligands decompose, in order to gain information on the effect of the metal ion on the ligand.

Apparatus for thermogravimetric analysis has been assembled and tested. Preliminary experiments on some test compounds (metal urea and oxime complexes) have been carried out in air in order to establish optimum heating rates etc. for reproducible measurements. A system for vacuum and inert atmosphere operation is in the early stages of construction.

We plan to complete the thermogravimetric analysis apparatus and commence serious study of some of our test compounds during the next semester.

Thermodynamics and Kinetics Vaporization Processes for Inorganic Materials — 470.35

Senior Investigator: H. Wiedemeier, Ph.D.
Assistant Professor of Chemistry

Research Staff: G. Sigai,
Undergraduate Assistant

The objectives of the present research project are the investigation

of the thermodynamic and kinetic properties of transition metal and group VI compounds upon vaporization at elevated temperatures.

For this purpose several special transport furnaces have been constructed which will be used for the preparation of single crystals. The furnaces are now being installed and calibration will start soon. A manifold for vaporization studies is in the construction stage, due to the delayed move to the new IMRC building.

Preparation of some sulfides and selenides is planned for the future.

Spectroscopic Studies of Synthetic Reversible Oxygen-Carrying Chelates 470.36

Senior Investigator: S.E. Wiberley, Ph.D.
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The object of our work is to prepare and to study solid iron (II) dimethylglyoxime ($Fe(dmg)_2$) as a possible synthetic reversible oxygen carrying chelate. Of special interest is the effect of such oxygen exchange on the infrared spectrum of the compound, for such a study might provide information on the nature of the structure of the chelate and of the bonding of oxygen to it.

The precursors of $Fe(dmg)_2$, $Fe(dmg)_2(py)_2$ (py = pyridine) and $Fe(dmg)_2 \cdot 2NH_3$ have been prepared by several alternate synthetic procedures. Gravimetric iron analysis have been carried out on all products and their infrared spectra have been measured.

Removal of base by thermal methods from the precursors to yield $Fe(dmg)_2$ has proved to be quite difficult and has hampered work in studying possible reversible oxygen exchange of these compounds. Future research efforts will be directed toward experimentation with other preparative techniques and the development of methods for removal of base molecules from these precursors. X-ray diffraction analysis will also be employed to study these compounds and methods for elucidating their oxygen carrying properties developed.

TECHNICAL PAPERS SUBMITTED FOR PUBLICATION

Reynolds, J., Sternstein, S.S.
"Effect of Pressure on the Infrared Spectra of Some Hydrogen-Bonded Solids"
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Safrany, D.R., Reeves, R.R., Harteck, P.
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Published by Butterworth as a book. Reprints sent to NASA 11/17/64

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"Experimental X-ray Stress Analysis Procedures for Ultra-High Strength Materials"
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Submitted to Physical Review

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"Thermodynamics of Crystalline Linear High Polymers V. Extended-Chain Copolymers of Polyethylene"
Submitted to Journal of Polymer Science

Wunderlich, B.
"Specific Heat of Polyethylene Single Crystals"
Submitted to Journal of Physics Chemistry

Wunderlich, B.
"Zeitabhangige Vorgänge des Kristallisierens und Erstarrens bei Linearen Hochpolymeren"
Submitted to Die Kunststoffe

Vogt, Jr., L.H., Katz, J.L., Wiberley, S.E.
"The Crystal and Molecular Structure of Ruthenium-Sulfur Dioxide Coordination Compounds, I. Chlorotetraammine (sulfur dioxide)-ruthenium (11) chloride"
Submitted to Journal of Inorganic Chemistry

Vogt, Jr., L.H., Katz, J.L., Wiberley, S.E.
"X-Ray and Infrared Studies of Several Ruthenium-Sulfur Dioxide Complexes"
Submitted to Journal of Inorganic Chemistry

APPENDIX A

Members of Interdisciplinary Materials Research Center
Faculty Committee

S.E. Wiberley, Chairman	Professor of Analytical Chemistry and Dean of the Graduate School
G.S. Ansell	Associate Professor of Metallurgical Engineering
W.H. Bauer	Professor of Physical Chemistry and Dean of the School of Science
A.A. Burr	Professor of Metallurgical Engineering and Dean of the School of Engineering
R.H. Hartigan	Director of the Research Division
H.B. Huntington	Professor of Physics, Chairman of the Department of Physics
G.J. Janz	Professor of Physical Chemistry Chairman of the Department of Chemistry
F.V. Lenel	Professor of Metallurgical Engineering
S.S. Sternstein	Assistant Professor of Chemical Engineering
J.M. LoGiudice	Administrative Director of the Interdisciplinary Materials Research Center